Viscoelastic properties of orthodontic adhesives used for lingual fixed retainer bonding

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Abstract: OBJECTIVE: To evaluate the viscoelastic properties of two experimental BPA-free and one BisGMA-based orthodontic resin composite adhesives for bonding fixed retainers. METHODS: A commercially available BisGMA-based (TXA: Transbond LR) and two bisphenol A-free experimental adhesives (EXA and EXB) were included in the study. The viscoelastic behavior of the adhesives was evaluated under static and dynamic conditions at dry and wet states and at various temperatures (21, 37, 50℃). The parameters determined were shear modulus (G), Young's modulus (E) under static testing and storage modulus (G1), loss tangent (tanδ) and dynamic viscosity (n*) under dynamic testing. Statistical analysis was performed by 2-way ANOVA and Bonferroni post-hoc tests (α=0.05). RESULTS: For static testing, a significant difference was found within material and storage condition variables and a significant interaction between the two independent variables ($p<0.001$ for G and E). EXA demonstrated the highest G and E values at 21℃/dry group. Dry specimens showed the highest G and E values, but with no significant difference from 21℃/wet specimens, except EXA in G. Wet storage at higher temperatures (37℃ and 50℃) adversely affected all the materials to a degree ranging from 40 to 60% ($p<0.001$). For dynamic testing, a significant difference was also found in material and testing condition groups, with a significant interaction between the two independent variables ($p<0.001$ for G1 and n*, $p<0.01$ for tanδ). Reduction in G1, and n* values, and increase in tanδ values were encountered at increased water temperatures. SIGNIFICANCE: The apparent detrimental effect of high temperature on the reduction of properties of adhesives may contribute to the loss of stiffness of the fixed retainer configuration under ordinary clinical conditions with unfavorable effects on tooth position and stability of the orthodontic treatment result.

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Viscoelastic properties of orthodontic adhesives used for lingual fixed retainer bonding

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Short title: viscoelastic properties of orthodontic adhesives

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ABSTRACT

Aim: To evaluate the viscoelastic properties of two experimental BPA-free and one BisGMA-based orthodontic resin composite adhesives for bonding fixed retainers.

Materials and Methods: A commercially available BisGMA-based (TXA: Transbond LR) and two Bisphenol A-free experimental adhesives (EXA and EXB) were included in the study. The viscoelastic behavior of the adhesives was evaluated under static and dynamic conditions at dry and wet states and at various temperatures (21,37,50^{o}C). The parameters determined were shear modulus (G), Young's modulus (E) under static testing and storage modulus (G_1), loss tangent (tan δ) and dynamic viscosity (n*) under dynamic testing. Statistical analysis was performed by 2-way ANOVA and Bonferroni post-hoc tests (α=0.05).

Results For static testing, a significant difference was found within material and storage condition variables and a significant interaction between the two independent variables ($p<0.001$ for G and E). EXA demonstrated the highest G and E values at 21^{o}C/dry group. Dry specimens showed the highest G and E values, but with no significant difference from 21^{o}C/wet specimens, except EXA in G. Wet storage at higher temperatures (37^{o}C and 50^{o}C) adversely affected all the materials to a degree ranging from 40-60% ($p<0.001$). For dynamic testing, a significant difference was also found in material and testing condition groups, with a significant interaction between the two independent variables ($p<0.001$ for G_1 and n^*, $p<0.01$ for tan δ). Reduction in G_1 values (37-40% at 37 ^{o}C and 52-59% at 50^{o}C), reduction in n^* values (25 to 31% at 37^{o}C and 34 to 42% at 50^{o}C) and increase in tan δ values (125-150% at 37^{o}C and 133-250% at 50^{o}C) were encountered at increased water temperatures.

Clinical significance The apparent detrimental effect of high temperature on the reduction of properties of adhesives may contribute to the loss of stiffness of the fixed

retainer configuration under ordinary clinical conditions with unfavourable effects on tooth position and stability of the orthodontic treatment result.

INTRODUCTION

Prevention of relapse is a major issue in orthodontic therapy and often requires long-term retention with preferably fixed retainers that require minimal patient compliance [1,2]. From the various types of fixed retainers described in the literature, those consisting of braided or solid metallic wires bonded to enamel with resin composite orthodontic adhesives are the most frequently used, despite the recent developments in resin impregnated polymer- and glass-fibers [3]. The intraoral performance of such systems composed of heterogeneous materials mainly depends on the strength parameters of the weakest part, the orthodontic adhesive resin, which dominates the stress transfer characteristics of the device to the bonded teeth and also demonstrates the highest failure incidence of the components involved [4].

Orthodontic resin composite adhesives for lingual retainer bonding are conventional particle-filled composites of medium to high filler content. These materials have been subjected to property modifications including viscosity optimization to reduce free flow, surface tension adjustment for adequate wetting of enamel and wire surfaces, softer consistency than highly filled materials for easy wire entanglement with enamel and thixotropic behavior with high recovery rates after shear thinning to ensure precise application. Orthodontic resin composite adhesives, like the restorative resin composites, demonstrate time-dependent mechanical properties [5]. Therefore, characterization of their viscoelastic behavior can help in understanding their performance under static and dynamic loading. Parameters such as flexural and shear modulus, loss tangent and dynamic viscosity show the ability of the polymers to withstand stresses and to recover during the unloading phase (elastic or inelastic/irreversible strain) under various testing conditions (*i.e.*, different temperatures, presence of water) [5,6]. This is more important when materials free of

bisphenol-A (BPA) derivatives are designed as alternatives to the commonly used BPA derivatives (BisGMA, BisEMA, BisDMA etc) to reduce the possible exposure to BPA release and the associated biological hazards [7,8]. Nevertheless, the stiff bisphenol aromatic backbone of BisGMA-type monomers, highly contributes to the rigidity of the final material and hence cannot be easily replaced in dental resin composite technology [9].

Despite their clinical significance, viscoelastic properties have not been thoroughly studied in orthodontic adhesives. Instead, most experimental research is focused on bracket bonding to enamel and restorative materials. In the present study, a well-established technique was used to evaluate the viscoelastic properties of two experimental BPA-free and one BisGMA-based orthodontic adhesives for bonding fixed retainers and a conventional flowable restorative liner. The null hypothesis was that there are no statistically significant differences in the viscoelastic properties among the materials selected under the experimental conditions used.

MATERIALS AND METHODS

The materials tested and their composition are summarized in Table 1. The mechanical properties of the orthodontic adhesives were investigated under static and dynamic testing. The specimens were prepared by inserting the adhesive into glass capillary tubes (Ø = 1 mm, L = 18 mm, n = 4 per product and testing condition) and thoroughly light curing by two 20-s sequential overlapping light exposures employing a LED curing unit (Bluephase G2, Ivoclar Vivadent, Schaan, Liechtenstein) emitting 1200 mW/cm^2 light intensity at the wavelength region 500-400 nm.

The method used in the present study has been successfully employed for determination of the viscoelastic behavior of resin composite samples under creep,

constant load rate, resonant and subresonant dynamic experiments in both torsion and flexure [6]. The apparatus utilized is described in Fig 1. Each specimen was mounted between a 0.5 mm thick plexiglas disc and a rod by using a centering jig. A high intensity permanent Sm-Co magnet (M = 1.12 x 10^{-2} Nm/A) with a thin mirror (Ø = 1.55 mm) bonded to the magnet, was attached at the end of each specimen, and the assembly was placed at the center of a Helmhotz coil. The weight of the magnet caused only a minor constant axial tensile stress with no constraints on specimen torsion or extension. The torque on the specimen was controlled by the current in the coil. The spot of a He-Ne laser beam reflected by the mirror was traced onto a calibrated chart placed at a distance D = 944 cm and the rotation angle of the mirror (φ) was calculated from the displacement of the laser beam on the chart (X) by the equation (Eq. 1):

$$\varphi = 2X/D. \quad (1)$$

The materials were tested after 24 h storage under the following conditions: i) Dry at 21°C, ii) immersed in water at 21°C, iii) immersed in water at 37°C and iv) immersed in water at 50°C. The conditions were controlled by placing a thin plastic tube (Ø = 16 mm, L = 18 mm) over the specimen which was attached to the disk, creating thus a water containing chamber capable of temperature control (± 0.5°C) via a heating element and a thermocouple.

Under static testing a constant torque was applied to each specimen for 10 s and then instantly released with the angular displacement being recorded. Depending on the alignment of the coil the specimen was tested either under torsion or bending. In the former case, shear modulus is the ratio of shear stress to shear strain ($G = \sigma/\gamma$) and was calculated from the equation (Eq. 2):

$$G = 2ML / \pi r^4 \varphi \quad (2)$$

where r is the specimen radius, L the length and M the magnet torque. In the case of bending, the coil was rotated for 90°. Young's modulus *E* was calculated by the ratio of flexural stress to flexural strain $(E = \sigma/\varepsilon)$, which for cylindrical specimens is given by the equation (Eq. 3):

$$E = 64ML / \pi d^4 \varphi ,$$

where d is the diameter of the specimen.

For dynamic testing, frequencies ranging from 1 to 150 Hz were applied to the specimens. A function generator connected to the Helmholtz coil created a sinusoidal torque. The displacement or amplitude was measured on the chart for each frequency. The viscoelastic properties were calculated from the resonance frequency v_0, corresponding to the peak amplitude and also from the resonance full width Δv, which is the difference between the two frequencies at which the amplitude is half of the maximum.

In the confines of linear viscoelasticity, stress and strain vary sinusoidally. Storage modulus (G_1) is in-phase with strain, while loss modulus G_2 (related to the dissipation of energy) is 90° out-of-phase with strain. In stiff solids the complex modulus G^* is almost equal in magnitude to the storage modulus G_1, because G_2 is small when compared to G_1. Storage modulus is given by the equation (Eq. 3):

$$v_0 = (1/2\pi) \times (G_1 \pi r / 2LI)^{1/2} ,$$

with r and L being the radius and length of the specimen and I the moment of inertia of the magnet.

The ratio of the imaginary part to the real part (G_2/G_1) of the complex modulus G* is the loss tangent (tan δ) that expresses the phase angle between stress and strain sinusoids. Loss tangent is proportional to the energy loss per cycle and is given by the equation (Eq. 4):

$$\tan \delta = (1/3^{1/2}) \times (\Delta v / v_0).$$

Dynamic viscosity was calculated from (Eq. 5):

$$n^* = (1/2\pi v_0) \times (G_1^2 + G_2^2)^{1/2}.$$

Statistical analysis was performed by two-way ANOVA and Bonferroni post-hoc tests, with material and testing condition as discriminating variables. A 95% confidence level ($\alpha = 0.05$) was selected. For the statistical analysis the Sigma Stat (v. 3.1, Jandel, S. Raphael, CA, USA) software was used.

RESULTS

The results of static properties are summarized in Table 2. There was a statistically significant difference within the material and testing condition independent variables ($p<0.001$ for G and E) and a significant interaction between the two independent variables ($p<0.001$ for G and E). EXA demonstrated the highest G and E values at 21°C/dry group. Dry specimens showed the highest G and E the values, but with no significant difference from 21°C/wet specimens, except EXA in G. Nevertheless, wet storage at higher temperatures (37°C and 50°C) adversely affected all the materials. The reduction in G and E mean values ranged from 34 to 41% at 37°C and 54 to 62% at 50°C, respectively. At 37°C and 50°C, no statistically significant difference was found between EXA and TXA. The G values of EXB were the lowest under all storage conditions. EXA showed the highest E values under all storage conditions, followed by TXA and EXB.

The results of dynamic properties are presented in Fig. 2. Again a statistically significant difference was found in material and testing condition groups ($p<0.001$ for G_1 and n^*, $p<0.01$ for tan δ) with a significant interaction between the two independent variables ($p<0.001$ for G, n^* and $p<0.01$ for tan δ). Dry specimens

showed no significant differences from 21ºC/wet specimens in G_1 (except EXA), n^* and tan δ (except TXA). All the dynamic properties tested were strongly affected after wet storage at 37ºC and 50ºC. Reduction in G_1 values (37 to 40% at 37ºC and 52 to 59% at 50ºC), reduction in n^* values (25 to 31% at 37ºC and 34 to 42% at 50ºC) and increase in tan δ values (125-150% at 37 ºC and 133-250% at 50 ºC) were encountered at increased water temperatures. No significant differences were found between EXA and TXA in G_1 (37ºC/wet and 50ºC/wet), n^* (37ºC/wet) and tan δ (37ºC/wet and 50 ºC/wet). EXB exhibited the lowest values in G_1 and in n^*, except from TXA at 37ºC/wet group. The tan δ values reached a plateau after 21ºC/wet storage in TXA, but were constantly increasing in EXA and EXB, reaching those of TXA at 50ºC/wet group.

DISCUSSION

The results of the present study revealed significant differences in the static and dynamic properties of the orthodontic adhesives tested, which were influenced by the experimental conditions used. Therefore, the null hypothesis should be rejected.

The technique used in the present study apart from composite restoratives has already been employed for assessment of resin luting agents, impression materials, fiber-reinforced posts and dentine adhesives [10]. All the orthodontic adhesives tested demonstrated viscoelastic behavior, as they stored energy during their deformation. The values obtained for G and E under static loading were at the level previously reported for bulk-fill liners, that are below the values of the main bulk-fill restoratives [5]. This difference should be attributed to the lower filler content of the orthodontic adhesives, in order to provide proper viscosity and handling properties. However, EXA resulted in G and E values comparable to several conventional restorative

composites [6], although the filler content of the material was lower than the control (TXA). A possible explanation is the increased C=C conversion of the polymer network in EXA comparison with TXA [11], due to the lower molecular weight of the monoaromatic dimethacrylate monomer PGDMA, in comparison with the high molecular weight bis-aromatic BisGMA and the associated steric hindrance effects induced by the latter. Nevertheless, the critical role of the filler content in G and E for these systems is profound [12], considering that EXB, which demonstrated the lowest G, E and G_1 values in the present study, resulted in the highest conversion in comparison with EXA and TRX [11].

Testing at 21ºC in dry and wet environment may provide a means of understanding the effect or water plasticization under isothermal conditions. Wet storage at 21ºC did not induce significant reduction in G and G_1 in the materials, except EXA, although E was not affected. This may be explained by softening of the shear modulus. Water storage at higher temperatures (37ºC and 50ºC) strongly affected G and E values apparently due to excessive resin softening. This may have a detrimental effect at bonded regions with high stress concentration. It is quite interesting that the percentage of reduction in both G and E was approximately 40% after storage at 37ºC and 60% after storage at 50ºC. Apparently the increased molecular mobility of hot water may enhance the plasticization effect in shear (G) and bending (E) moments.

The results of dynamic testing demonstrated a ranking of G_1 similar to G. The same reduction profile was observed in the n^*. The values after wet storage at 37ºC and 50ºC were quite low and may indicate an excessive in service viscous flow, which may affect the stress transfer characteristics of the retainers to the resin-enamel interface. The tan δ parameter, defined as the ratio of lost to stored energy in cyclic

deformation, was rapidly increased in TXA after storage in water (21°C/dry), whereas in EXA and EXB the corresponding values resembled those of the dry controls. Considering that the dry tan δ values of all the adhesives showed no statistically significant difference, it follows that TXA was more sensitive to water than the testing temperature, contrary to EXA and EXB where the tan δ values were more temperature dependent. Materials with high tan δ values show a delayed elastic response to strain and therefore the energy lost in each is transformed to viscous flow and heat.

Regarding the clinical significance of the results, it should be noted that while in restorative composites dynamic testing may be more relevant because of the nature of the stresses under masticatory cycles, this may not be the case in orthodontic adhesives. When these materials are used for bracket bonding, they are loaded under constant stress upon wire activation, which decays slowly with tooth movement. Therefore, static testing may be considered as more reliable for this application. Nevertheless, for lingual retainers masticatory cycles may expose the adhesive materials to dynamic loading conditions under a complex loading pattern affected by the occlusion characteristics and masticatory forces [13]. The latter have been shown to be highly dependent on the pattern of craniofacial growth of patients. In general, in vertically excessive facial types characterized by long faces, forces are much higher than horizontal facial types, which are usually present in square faces. The magnitude of forces applied in the anterior part of dentition, measured at one point of the incisal edge of the mandibular incisor has been estimated to vary between 100 and 200 N [11,14,15]. Although these values derived from human subjects and with the use of stain gauges, the actual biting stress and associated loads exerted in the anterior dentition is expected to be much lower than the reported values owing to the distribution of the loads to a much larger area than the mandibular incisor incisal edge

utilized in this study. A study attempted to assess the effect of loading during mastication of arch configured lingual fixed retainers *in vitro*, demonstrated that residual forces and moments were exerted after 15 N unloading, which implies that the evaluated fixed retainers were not passive after *in vitro* vertical loading, even at loading with forces much lower than the reported [16]. This fact may explain the unexpected movements of teeth bonded on fixed retainers detected long-term in vivo. This type of movement cannot be considered as relapse, since teeth have been reported to move on a direction opposite to the pre-treatment condition, thereby establishing the role of lingual fixed retainer as a non-passive, tooth-moving mechanism [17,18]. The results of the present investigation provide further support to this adverse action of retainers focusing on the involvement of adhesives in this phenomenon, through the reduction in their stiffness documented to occur at higher temperatures, which however, are within the temperature range found in routine conditions [19].

Within this context, it was demonstrated that, at ambient temperature, the properties affected by water storage were G (in EXB) and tan δ (in TXA). However, upon temperature increase the properties were strongly affected, which implies an increasing failure propensity. From the materials tested, the BPA-free EXA provided similar or superior results than the control (TXA), which is based on BPA components and therefore may be considered as a promising alternative for TXA [20]. On the other hand, the performance of EXB was ranked as significantly inferior to TXA and EXA in static and dynamic testing. Further research should be done to document the clinical relevance of these findings.

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Table 1. The orthodontic adhesives tested

MATERIAL	COMPOSITION	MANUFACTURER
Transbond-LR Adhesive CODE: TXA	Resin : BisGMA, TEGDMA, Catalysts : Dimethylbenzocaine, Diphenyliodonium hexafluorophosphate, Filler : Silanated quartz, silnated silica (75-85% wt)	3M ESPE, St. Paul, MN, USA
Experimental Adhesive I CODE: EXA	Resin: PGDMA, TEGDMA, UEDMA, Catalysts: CQ, DEAEMA Filler: Silanated glass (70 wt%)	---
Experimental Adhesive II CODE: EXB	Resin: TEGDMA, UEDMA, Catalysts: CQ, DEAEMA Filler: Silanated glass (60 wt%)	---

BisGMA: Bisphenol glycidyl dimethacrylate, PGDMA: Phenyl carbamoyloxy-propane dimethacrylate, TEGDMA: Triethyleneglycol dimethacrylate, UEDMA: Aliphatic urethane dimethacrylate, CQ: Camporquinone, DEAEMA: Dimethylamino ethyl methacrylate

Table 2. Results of static testing (means and standard deviations). Same superscript letters indicate mean differences with no statistically significant differences ($p>0.05$) between conditions within each material group (capital case) and between material between material groups per condition (small case) groups per condition (small case).

STATIC PROPERTIES	TESTING CONDITIONS	TXA	EXA	EXB
Shear Modulus G (GPa)	21ºC/dry	4.82 (0.06) ^{A,a}	5.45 (0.04) ^{D,b}	2.67 (0.01) ^{H,c}
	21ºC/wet	4.73 (0.04) ^{A,d}	5.01 (0.08) ^{E,d}	2.67 (0.04) ^{H,e}
	37ºC/wet	2.95 (0.07) ^{B,f}	3.21 (0.06) ^{F,f}	1.68 (0.08) ^{I,g}
	50ºC/wet	2.24 (0.09) ^{C,h}	2.09 (0.08) ^{G,h}	1.09 (0.11) ^{J,i}
Flexural Modulus E (GPa)	21ºC/dry	11.7 (0.09) ^{A,a}	15.2 (0.07) ^{D,b}	8.83 (0.06) ^{G,c}
	21ºC/wet	11.7 (0.11) ^{A,d}	15.2 (0.09) ^{D,e}	8.71 (0.08) ^{G,f}
	37ºC/wet	7.71 (0.15) ^{B,g}	10 (0.11) ^{E,h}	5.6 (0.11) ^{H,i}
	50ºC/wet	4.48 (0.08) ^{C,j}	6.43 (0.08) ^{F,k}	4.63 (0.14) ^{H,j}

FIGURE LEGENDS

Fig. 1. Schematic appearance of the principles of function of the apparatus used for static and dynamic testing.

Fig. 2. The results of the dynamic properties G_1, n^* and tan δ.

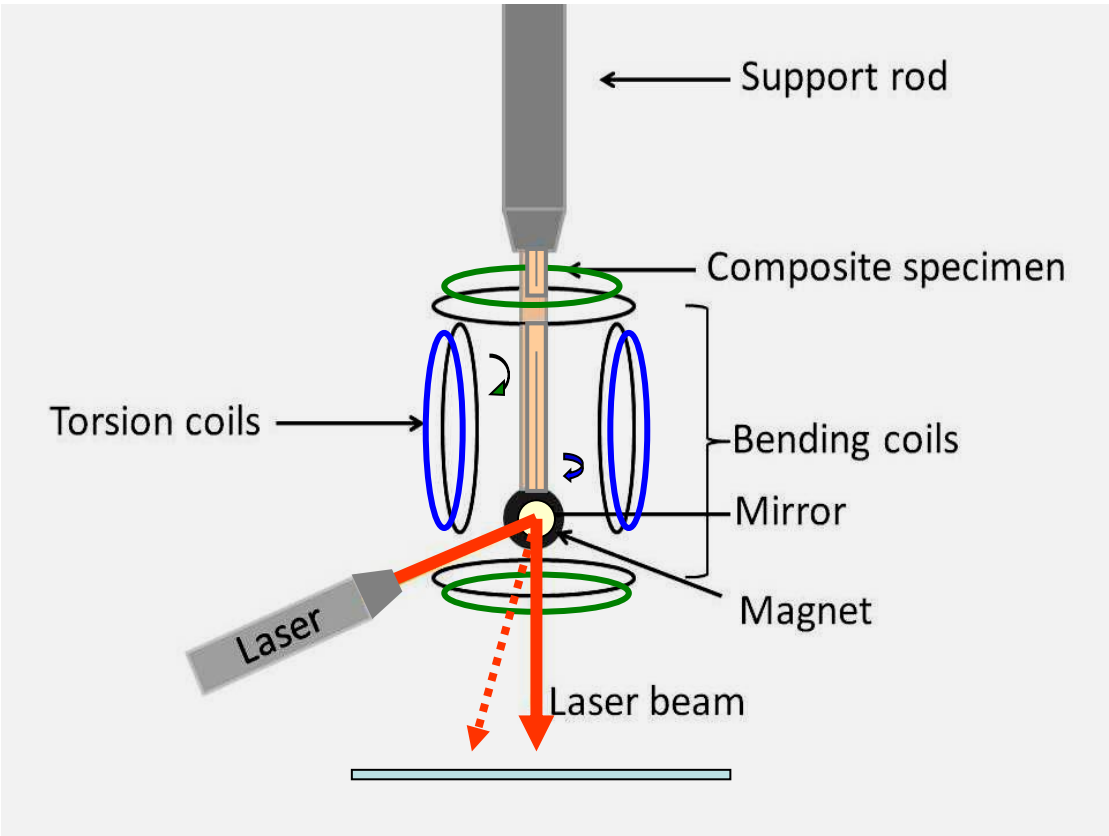
Support rod
Composite specimen
Torsion coils
Bending coils
Mirror
Magnet
Laser
Laser beam

Fig. 1

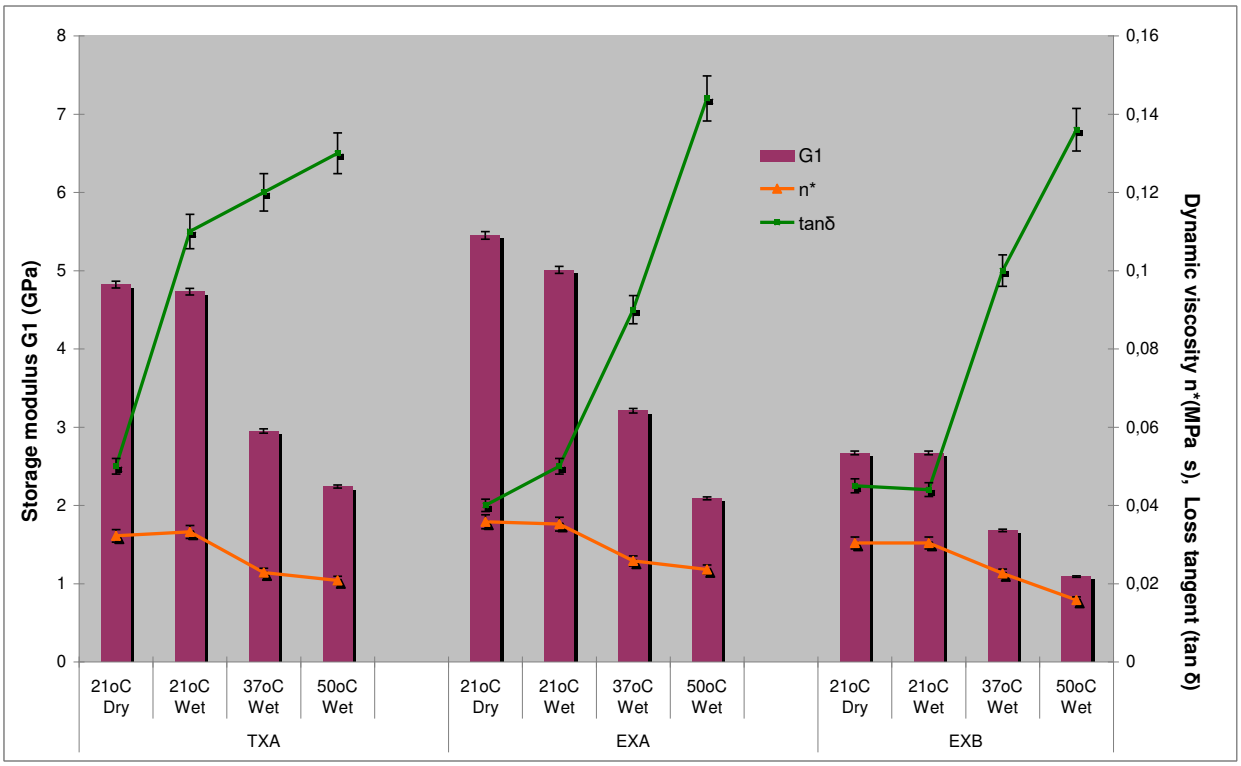
Storage modulus G1 (GPa)
Dynamic viscosity η*(MPa s), Loss tangent (tan δ)
G1
n*
tanδ
21oC Dry
21oC Wet
37oC Wet
50oC Wet
TXA
EXA
EXB

Fig. 2